INDUSTRIAL PILOT PROJECT

X-ray spectroscopy and tomography for improved understanding of metal-graphene composite behaviour and properties

THE INDUSTRIAL CHALLENGE

Thanks to its remarkable mechanical, electrical and thermal properties, graphene can reinforce metals and therefore replace scarce/critical metals with more common ones. Graphmatech produces metal-graphene composites for *e.g.* improved electrical contacts and corrosion barriers. To understand and to be able to predict how and why graphene enhances other material's properties, a deeper knowledge about the graphene-metal interaction is essential. Also, a deeper knowledge regarding the quality of resulting parts, when the material is used as powder for 3D-printing is of high value.

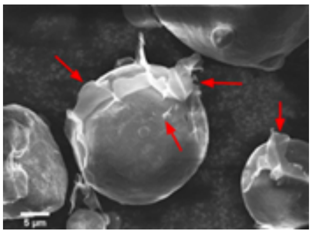


Figure 1. SEM-image of Cu-graphene particles with arrows indicating the ~5μm graphene flakes.

WHY USING A LARGE SCALE FACILITY

1. Electrical measurements can give information of the overall conductivity of metal-graphene composites, while angular resolved photoelectron spectroscopy (ARPES) allows confirmation of doping of graphene by direct measurements of the occupied part of the electronic band structure.

2. Potential carbide formation, which strongly affect e.g., the corrosion properties, can be studied by lab-based photoelectron spectroscopy, but the required removal of surface oxides by sputter etching is likely to damage the sample. The possibility to penetrate deeper into the bulk using hard x-ray photoelectron spectroscopy (HAXPES) allows non-destructive information.

3. Possible voids, defects and density differences in 3D-printed materials can be studied destructively using light microscopy, and for very small (<0.5mm) samples also using lab-based x-ray diffraction techniques. Synchrotron-based x-ray tomography can give a 3D-image in a non-destructive way.

HOW THE WORK WAS DONE

1. To study a single graphene flake (~5μm) using nano-ARPES at the ANTARES beamline of Soleil, France (support P. Dudin, J. Avila), the copper(Cu)-graphene powder was pressed into a foil of gold.

2. Carbide formation was studied in 3D-printed and polished pieces (8x8x5mm) of Cu and steel (316L) with and without graphene, using HAXPES at the SPLINE beamline, ESRF, France, using a photon energy of 9 keV (support J. Rubio Zuazo).

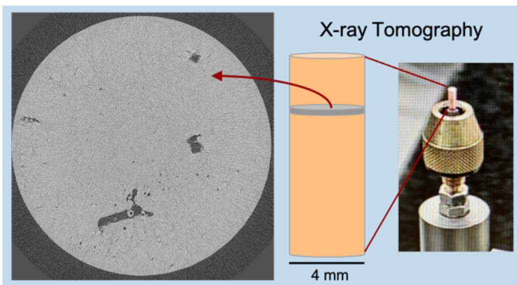
3. Finally, a printed piece of copper(Cu)-graphene, shaped to a thin rod (4mm diameter, 50 mm long), was studied using x-ray tomography in transmission mode at the P21.2 beamline of Petra III, Germany (support U. Lienert, DESY).

THE RESULTS AND EXPECTED IMPACT

1. The lateral resolution of 1μm of the ANTARES beamline allowed detection of a single graphene flake (~5μm) attached to a Cu particle. To perform ARPES and thereby detect the occupation of the valence and conduction bands is therefore, in principle, possible, but was not feasible within the 24h available. The method require detection of one single flake and x-rays are difficult to focus narrower than 5μm.

2. The HAXPES measurements indicate partially carbide formation in 316L/graphene but not in Cu/graphene, and this method will be further explored.

3. X-ray tomography easily gave a clear view of voids down to 10μm and their spreading in 3 dimensions. This can be useful for additive manufacturing of small components of approximately 5 mm in size.



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GRAPHMATECH

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